Optical Properties of Semiconductor-Metal Composite Thin Films in the Infrared Region

by

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ABSTRACT

Germanium:Silver (Ge:Ag) composite thin films having different concentrations of Ag, ranging from 7% to 40% have been prepared by dc co-sputtering of Ge and Ag and the films' surface morphology and optical properties have been characterized using transmission electron microscopy (TEM) and infrared spectrophotometry. It is seen that while the films containing lower concentrations of Ag have island-like morphology (i.e. Ag particles distributed in a Ge matrix), the higher metallic concentration films tend to have symmetric distribution of Ag and Ge. The optical constants (i.e. refractive index n and absorption index k) derived from the measured optical properties show a semiconductor behavior even up to 40% concentrations of Ag, beyond which the metallic properties dominate over the entire infrared spectrum. Comparison of the *n* and k data with the two well known effective medium theories, namely, the Maxwell-Garnet theory and the Bruggeman theory, shows that either of these theories has limited scope in predicting the optical properties of semiconductor-metal composite films in the infrared region. However, an empirical polynomial equation can simulate the experimental data at all wavenumbers of the IR spectrum.

Key words: Optical, Dielectric, Inhomogeneous, Infrared, Effective Mean Field Theory, Composite Films, Germanium, Silver.

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1. Introduction

Present-day scientific and technological developments demand the use of novel optical materials which exhibit unique optical properties not observed in conventional materials. Investigations have focused on developing new optical materials and efficient ways of tailoring the properties of existing materials within the scope of the available preparation techniques. The latter approach is often simpler and easier to implement. To this end, thin film deposition techniques, namely, evaporation,[1,2] ion beam assisted,[3] and sputtering have been made use of to successfully prepare composite/inhomogeneous dielectric thin films.[1-4] This has resulted in thin films, having unique optical properties, which have found applications in optical thin film devices.[1,2,5] Similarly, dielectrics have been co-deposited with different metals leading to a whole family of cermet films[4,6,7] which have applications in solar energy conversion devices.[8,9] Both dielectric-dielectric and metal-dielectric composite films have been widely investigated with emphasis on the optical properties in the solar and near infrared spectral regions. However, there are neither any studies on the optical properties of these materials in the middle and far infrared (IR) regions, nor any attempt to find out the viable combinations of composite/inhomogeneous materials for the infrared region, except for a few remarks.[8] Nevertheless, a growing use of the IR spectrum for various scientific and technological missions either in space or ground,[11] demands the use of such novel materials. In view of this, the present investigation has been organized to systematically study the infrared optical properties of composite thin films.

In this paper, starting with a brief description of viable combinations of composite optical materials in the IR region, experimental results of the preparation and characterization of a few specific composite thin films are presented. The scope of the phenomenological and empirical theories to account for the experimental results is also discussed.

2. Infrared Composite Optical Thin Films

From a critical review of the optical properties of IR materials[10-12], it can be observed that there is a paucity of optical materials for the IR region, which are not only very good absorbers but also medium reflectors. Such interesting optical properties can be expected from semiconductor-metal composite materials for which many viable combinations such as Ge:Ag, Ge:Au, Si:Au and Si:Ag can be thought of. These materials can find applications as broad-band infrared absorbers in optical, opto-electronic and thermal control systems. In the present investigation, the preparation and characterization of the Ge:Ag combination in thin film form are discussed.

3. Preparation:

Composite films of Ge:Ag having uniform concentrations of Ag varying from 7% to 40% are prepared by dc magnetron co-sputtering of Ge and Ag. The sputtering process utilizes a 5 cm diameter planar 99.999% pure Ag target mounted on a dc sputter gun manufactured by U.S. Inc., and a two-piece 3.8 cm diameter doughnut shaped 99.9999% pure Ge target mounted on a Research S-Gun manufactured by Sputtered Films

Inc. The targets (from International Advanced Materials, Suffern, New York) are oriented at a 45° angle to the substrate plane to achieve a high degree of uniformity over the substrate geometry. Uncooled substrates are mounted at a distance of 15 cm from the targets. The sputtering chamber is pumped by a turbomolecular pump to a base pressure less than 10^{-4} Pa and back-filled with argon.

In the present set-up the concentrations of Ag and Ge are decided by the sputtering yield rates of Ag and Ge under a given set of experimental conditions, namely argon flow rate, input power and chamber pressure. The sputtering/deposition rates of Ag and Ge are arrived at by measurement of thicknesses of the films deposited over a known amount of time. The thicknesses are measured using a Tencor Alpha Talystep 250 Profilometer with a measurement accuracy better than 0.5 nm. Thus the sputtering set-up has been calibrated for different sputtering rates against input power. The deposition rate of Ge is about 0.12 nm/min under 4.4 Pa of argon pressure with 5 Seem of argon flow and 1 watt of power applied, where as the deposition rate of Ag is 0.5 nm/min for 1 watt of power. The input powers to Ge and Ag targets are maintained in such a proportion to obtain the desired metallic concentration/volume fraction.

For IR optical properties' characterization, films deposited on CdTe and soda lime glass are used. The films sputtered on thin soda lime glass microslide cover slips are used for TEM and stress pattern observations. It may be noted that in all the films, a neutral stress state has been achieved, indicating that the films are mechanically very stable.

4. Characterization:

The surface characterization of the thin films of Ge:Ag is carried out using transmission electron microscopy (TEM). The rear side of the coated thin microslide samples are ground to reduce the substrate thickness to a few microns after which they are etched in an argon ion beam milling system at 5kV and 0.5mA, with a 12° incident angle, to achieve electron transparency in a free standing film. The micro-samples are then transferred to a substrate holder for microscopic observation utilizing a Topcon 002B Transmission Electron Microscope.

The optical reflectance and transmittance of the samples are characterized using a Beckmann IR Spectrophotometer, Model 4800. The instrument is a priori calibrated for 100% transmittance, and reflectance against a standard Ge sample in the reflectance mode before carrying out transmittance and reflectance measurements of the samples respectively. For measurements of reflectance, a 10° specular reflectance accessory is made use of. From the measured reflectance and transmittance, the optical constants ,i.e. refractive index n and absorption index k, are deduced using an inverse method of synthesis[13,14] in which the R and T equations connecting the optical constants of the films to the measured optical properties, the thickness of the film and substrate optical constants, are solved by a numerical iteration technique. This procedure has been applied to determine the optical constants of films having metallic concentrations up to 25%.

For films, having 40% metallic content, which have hardly any transmission, a reflectance measurement-based spectrophotometric (abbreviated as RMSP) technique is adapted.[15] In this technique, the measured reflectance, R, from the virgin film, and R′, from the film deposited with a transparent layer, are made use of,[15,16] and the optical constants are evaluated.

In the present studies, ZnS deposited by thermal evaporation is utilized as the transparent layer over the opaque virgin film of interest. The refractive index of the ZnS layer is determined a priori over the IR spectrum from 4000 to 700 cm-′ and then the optical constants of the film are evaluated using the RMSP technique. However, to determine n and k from 700 cm-′ to 320 cm-′, theoretical simulation is carried out until $(R - R_{Th})^2 \leq 10^{-5}$. R_{Th}, the computed value of reflectance, for preset values of n and k is obtained from the equation

$$R_{Th} = \frac{[(n-n_o)^2+k^2]}{[(n+n_o)^2+k^2]} \tag{1}$$

where n and k are the optical constants of the opaque film and n_o is the refractive index of the medium.

It may be pointed out that to arrive at the n and k data for a given composite film, three films prepared under identical experimental conditions are characterized and the n and k data are evaluated by the appropriate techniques. The average of the three sets of data is taken as the experimental n and k data for a given composite film. The scatter in n and k over three sets of data are within 0.05 through out the IR spectrum.

5. Results and discussion:

5.1 Morphology:

The transmission electron micrographs of Ge:Ag composite films and the associated diffractograms are shown in Figs. la - lc. The calculated ratios of the observed d spacings in all the diffraction patterns agree with the standard d spacing ratios of Ag. Micrographs of films having higher concentrations of Ag (25% and 40%), exhibit d spacing images with a few crystalline defects such as stat'king faults. On the other hand, the Ge phase exhibits no crystalline features, in conformity with earlier investigations.[7] These observations clearly indicate that Ag crystallite are distributed in an amorphous Ge matrix. It is also obvious from the micrographs that the particle size and density of Ag increase as the concentration of Ag is increased. At lower concentration (13%), the particle size is as low as 20A, whereas they are in the range of 100-150A and 200-300Å for films having 25% and 40% concentrations of Ag, respectively.

It may be observed that at lower Ag content, Ag particles are sparsely distributed in the Ge matrix, which is similar to the morphology observed, in the case of Ni/Al_2O_3 composites, by Craighead, et. al.[6] However, at higher volume fractions of Ag, there is a tendency for a symmetric distribution of Ag and Ge, similar to those of Au/Al_2O_3 and Ag/MgO composite films.[7] In the light of these observations, it may be obvious to anticipate that the Maxwell-Garnet theory[18] should reasonably account for the optical properties of the Ge:Ag composite films having lower concentrations of Ag, and the Effective Medium Theory due to Bruggeman[19] which is based on a symmetrical distribution of the components of the composite materials, should adequately describe the optical properties of the composite films having higher concentrations of Ag (25% and 40%). These will be discussed in detail in the subsequent paragraphs.

5.2 Optical Properties:

The results of the optical constants n and k derived from the measured optical properties are presented in Figs. 2, 3, 4, and 5 for films having 7%, 13%, 25%, and 40% volume fractions of Ag, respectively. For the sake of convenience of presentation, these films are represented as F1, F2, F3, and F4. The measured optical properties R and T are also shown in Figs. 2a - 5a for comparison. It can be seen from the figures that the addition of Ag results in an increase of n and k. Even 7% of Ag is sufficient enough to increase n by 15% and the corresponding films have more than 30% absorption. This property can be exploited to produce high index Ge films with controlled absorption which can be utilized in many applications such as neutral density filters, beam splitters and semi-transparent high reflecting layers in Fabry-Perot etalons.

At lower metallic concentrations/volume fractions (FI and F2), the optical constants exhibit a weak dispersive behavior while films having higher volume fraction (F3 and F4) are highly dispersive in the spectral region below 1000 cm^{-1}. In addition to this, a resonance behavior centered between 700 and 500 cm^{-1} is also observed in F3 films, which is more pronounced at higher volume fractions. It is evident from Figs. 3 and 4 that the films, having as high as 25% of

Ag, retain the semiconducting behavior (n>k) with enhanced absorption. On the other hand, F4 films (40% of Ag) show a metallic behavior. The characteristic feature of metallic optical properties (i.e. k>n) is seen below 1000 cm^{-1}. Examination of the optical properties of other families of composite thin films, namely Ni/Al_2O_3, Au/Al_2O_3, Au/MgO, and Ag/Al_2O_3,[7] indicates that such a feature is rarely observed except in the case of Ag/Al_2O_3 having 60% of Ag. Thus, the exhibition of metallic-like optical properties at medium metallic concentrations may be unique to the family of semiconductor-metal composite systems.

In order to study the scope of the effective medium theories to explain the experimentally observed optical properties, theoretical calculations are carried out utilizing the associated equations from the Maxwell-Garnet theory (MGT)[18] and the Effective Medium theory (EMT) due to Bruggeman.[19] The deduction of n and k in the case of MGT is straight forward[20]. However, in the Bruggeman theory it involves either the solution of a quadratic equation for the average dielectric constant $<\epsilon>$[21], or a simultaneous non-linear equation which connects the real and imaginary parts of $<\epsilon>$ to the dielectric functions of the constituents. The simultaneous non-linear equation is solved by the Newton-Raphson iterative method[22]. Adapting the latter approach which is unambiguous and straight forward, the real and imaginary parts of the composite dielectric function are derived from which the n and k are evaluated. The Ge optical constants are set to 4.0 + j0.0 over the entire IR spectrum (4000 to 320 cm^{-1}) while the dielectric constant of Ag is evaluated by a Drude dielectric function[23],

$$\epsilon = -\frac{\omega_p^2}{\omega(\omega + j\omega_\tau)} \tag{2}$$

where ω_p is the plasma frequency and ω_τ is the inverse of the relaxation time.

While ω_p is set equal to $1.25 \times 10^4\ cm^{-1}$,[23] . ω_τ is given by $\omega_\tau = v_f/2\pi cr$ (v_f is the Fermi velocity of electrons in Ag, c is the velocity of light, and r is the radius of the Ag particles in the composite film) . By defining ω_τ in terms of v_f and r, the effect of reduced electron mean free path due to limited particle size is taken into account, the importance of which has been recognized by many investigators.[24-26] In the present calculations, r is obtained from the TEM analysis and v_f is set equal to 1.39×10^8 cm/sec.[27]

The results of the theoretical calculations are presented in the b and c components of Figs. 2 through 5, along with those of the experiments for easy comparison. From these figures it can be generally noticed that, irrespective of the nature of the composite film, either MGT or EMT (with one exception) predicts that the films have lower and lower absorption as the wavenumber decreases and become nearly transparent ($k < 0.05$) at the far infrared region, whereas experimentally the reverse is the case. The exception is that EMT predicts increase in n and k values with the decrease of wavenumber, for F4 films, which is in agreement with the experiment. The experimentally observed transition from semiconductor to metal in F4 films is also supported by the EMT results, even though the transition region and the optical constants do not match with the experimental results quantitatively. While the experiment predicts the transition at 1250 cm-′, the theory shows a broad transition region, spreading from 3000 to 1250 cm-′. It may be important to add that MGT does not indicate any such features. The success of EMT in explaining some of the features, at least qualitatively, may be attributed to the fact that EMT is not prejudiced toward a system of any particular composition and takes into account, in a mean field way, the interactions between the randomly dispersed constituent particles. On the other hand, MGT is confined to situations in which one component predominates in concentration over the other, making the theory inapplicable to F4 films. From the TEM studies presented earlier, it can be recalled that the F4 film exhibits a competitive morphology upon which EMT is developed.

A fair degree of agreement between theory and experiment can be observed in F1 films (7% cone. of Ag) , both in the case of MGT and EMT . The discrepancy is limited to only the far infrared region in which the films are more absorbing in practice. The agreement between theory and experiment is none too satisfactory in F3 films, whereas the degree of agreement in F2 films is in between that of F1 and F3. From the theoretical calculations and their comparison with experiments, it can be summarized that the two inhomogeneous theories, MGT and EMT, are not highly successful in explaining the features observed experimentally. This may not be totally unexpected, in the context of the results of the optical properties of composite films in the visible and infrared regions[6,7]. From these studies, it can be generally observed that for Au/Al_2O_3 and Ni/Al_2O_3 composite films having medium metallic concentrations (14% - 20%), k values predicted either by MGT or EMT are lower than the experimental values in the near-infrared region (10,000 - 3330 cm″′), whereas at higher volume fractions (26% in Au/Al_2O_3 and Ni/Al_2O_3), n and k predicted by EMT are higher than the experimental values. These discrepancies can also be seen in the present theoretical results.

In order to explain all the observed experimental results, a comprehensive approach is required in which there should be scope to consider the contributions not only from the individual components but also from the interactions between the metal and semiconductor. The mutual semiconductor-metal interaction may be responsible for dispersion of the optical constants and resonance behavior, predominantly seen in higher metallic concentration films and at the far infrared region.

In the absence of an appropriate theoretical scheme to precisely determine the optical constants of binary semiconductor-metal composite systems, an attempt has been made to determine the theoretical form of the equation which can yield the n and k values for any given volume fraction of metal and at any wavenumber.

For this, a polynomial equation of the following type is assumed,

$$\begin{pmatrix} n \\ k \end{pmatrix} = \sum_{i=1}^{m} \begin{pmatrix} P_i \\ Q_i \end{pmatrix} F^{i-1} \qquad (3)$$

where P_i and Q_i are the polynomial coefficients and F is the volume fraction/concentration of the metal in the composite thin film.

Using the experimental results of n and k at a given wavenumber and for different volume fractions, polynomial coefficients are determined by which the polynomial equation would give the best fit to the experimental results. For this purpose, a computer program based on the Vandermonde Matrix method is made use of.[28] Polynomial coefficients are determined for all wavenumbers starting from 4000 cm-' to 320 cm-'. It is seen that a fourth degree polynomial equation (m = 5) is able to satisfactorily account for the experimentally determined n and k data. Typical polynomial coefficients corresponding to different wavenumbers are given in Table I and the associated polynomial curves for n and k are presented in Figs. 6a and 6b respectively. This approach has been used to simulate the n and k of graded index semiconductor-metal composite films and study the optical properties of graded index composite films. These results will be presented in detail elsewhere[29].

6. Conclusions

Germanium: Silver (Ge:Ag) composite thin films have been successfully prepared by simple dc magnetron sputtering. The experimental studies indicate that Ge in combination with Ag produces films having a wide range of optical characteristics starting from low reflectance/absorptance to high reflectance/absorptance which can be controlled by metallic concentration. These films may have interesting applications such as selective infrared reflectors and high efficiency infrared absorbers.

The semiconductor-metal composite films give scope to study the mechanism of transition from semiconductor to metal. This type of transition of has been observed in relation to the optical properties at the infrared in the present investigation.

The available inhomogeneous theories (EMT and MGT) have limited scope to explain the observed features of the optical characteristics of semiconductor-metal composite thin films. A satisfactory explanation can emerge either by enlarging the scope of the existing theories or adapting a comprehensive approach which can take into account the possible interactions between the metal and semiconductor.

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TABLE I

Polynomial Coefficients for Ge:Ag Composite Films

WAVENUMBER cm^{-1}	P_1	P_2	P_3	P_4	P_5
4000	**4.0**	**15.04**	**-123.26**	**431.75**	**-495.96**
3000	**4.0**	**13.907**	**-85.799**	**239.85**	**-223.75**
2000	**4.0**	**10.879**	**-22.625**	**-45.026**	**157.66**
1000	**4.0**	**13.479**	**-47.007**	**81.651**	**-49.815**
700	**4.0**	**18.717**	**-87.051**	**219.38**	**-203.10**
400	3.997	25.439	-230.98	1351.1	-2223.0
WAVENUMBER cm^{-1}	Q_1	Q_2	Q_3	Q_4	Q_5
4000	0.000	S.2647	-2.4228	14.767	94.849
3000	0.000	6.9248	-44.800	284.23	-362.57
2000	0.000	5.0442	-20.396	181.78	-229.00
1000	0.000	3.7306	-12.045	286.09	‘426.99
700	0.000	10.687	-121.58	862.89	-1219.4
400	0.000	13.007	-116.81	587.89	‘571.81

Figure Captions:

Figs. 1a-1c: Transmission electron micrographs and the corresponding diffraction patterns (shown in the inset) for Ge:Ag composite films, 1a) 13% Ag; 1b) 25% Ag; and 1c) 40% Ag.

Fig. 2: Optical properties of Ge:Ag composite film with 7% Ag, 2a) Measured reflectance (R) and transmittance (T), 2b) Comparison of the experimentally deduced n and k with those from the MGT, 2c) Comparison of the experimentally deduced n and k with those from the EMT

Fig. 3: Optical properties of Ge:Ag composite film with 13% Ag, 3a) Measured reflectance (R) and transmittance (T), 3b) Comparison of the experimentally deduced n and k with those from the MGT, 3c) Comparison of the experimentally deduced n and k with those from the EMT

Fig. 4: Optical properties of Ge:Ag composite film with 25% Ag, 4a) Measured reflectance (R) and transmittance (T), 4b) Comparison of the experimentally deduced n and k with those from the MGT, 4c) Comparison of the experimentally deduced n and k with those from the EMT

Fig. **5**: Optical properties of Ge:Ag composite film with 40% Ag, 5a) Measured reflectance (R) and transmittance (T), 5b) Comparison of the experimentally deduced” n and k with those from the MGT, 5c) Comparison of the experimentally deduced n and k with those from the EMT

Fig. 6a: Variation of refractive index n of Ge:Ag composite films with respect to metallic volume fraction, at different wavenumbers, as calculated from a fourth degree polynomial equation

Fig. 6b: Variation of absorption index k of Ge:Ag composite films with respect to metallic volume fraction, at different wavenumbers, as calculated from a fourth degree polynomial equation

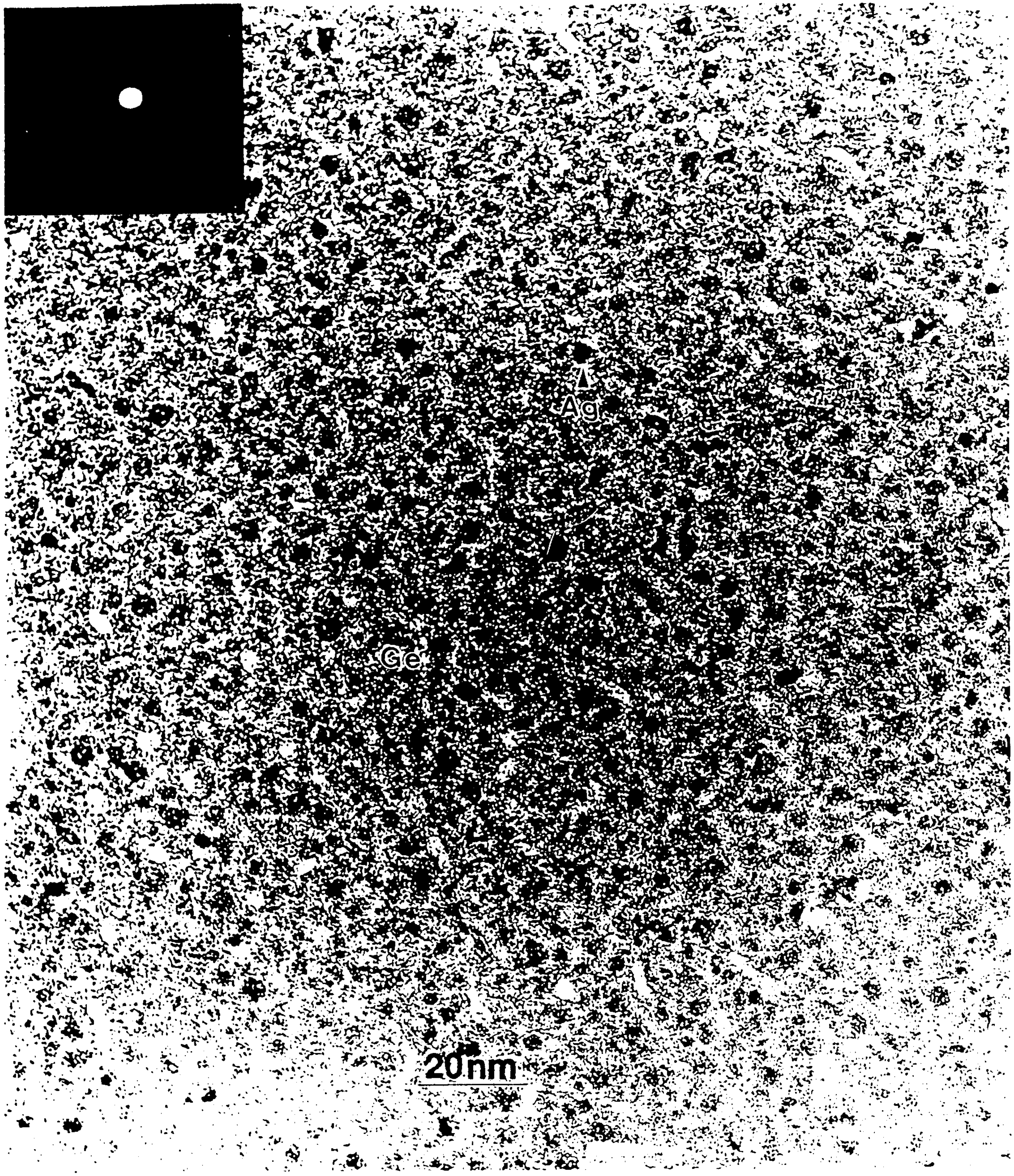


Fig 1a

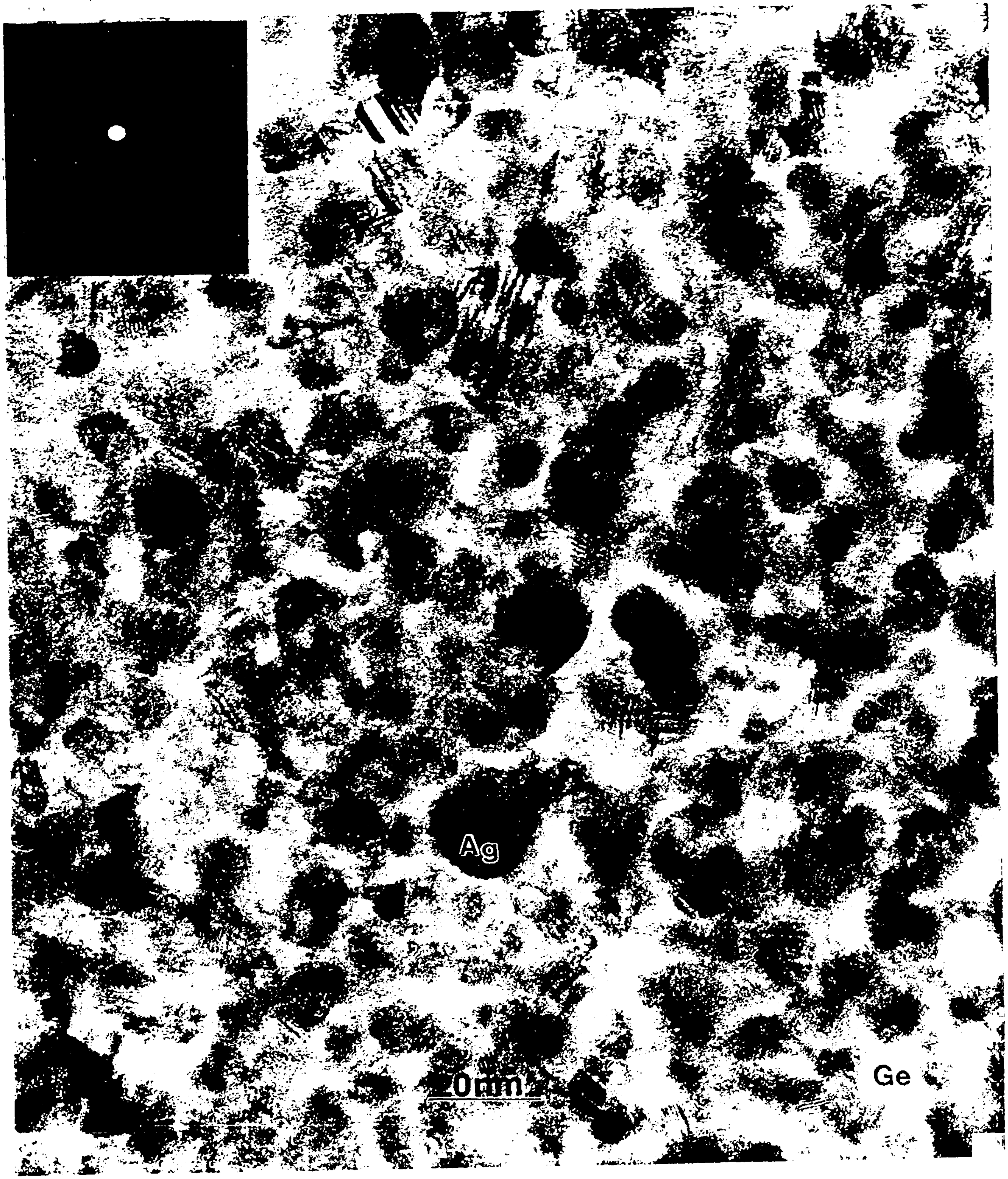
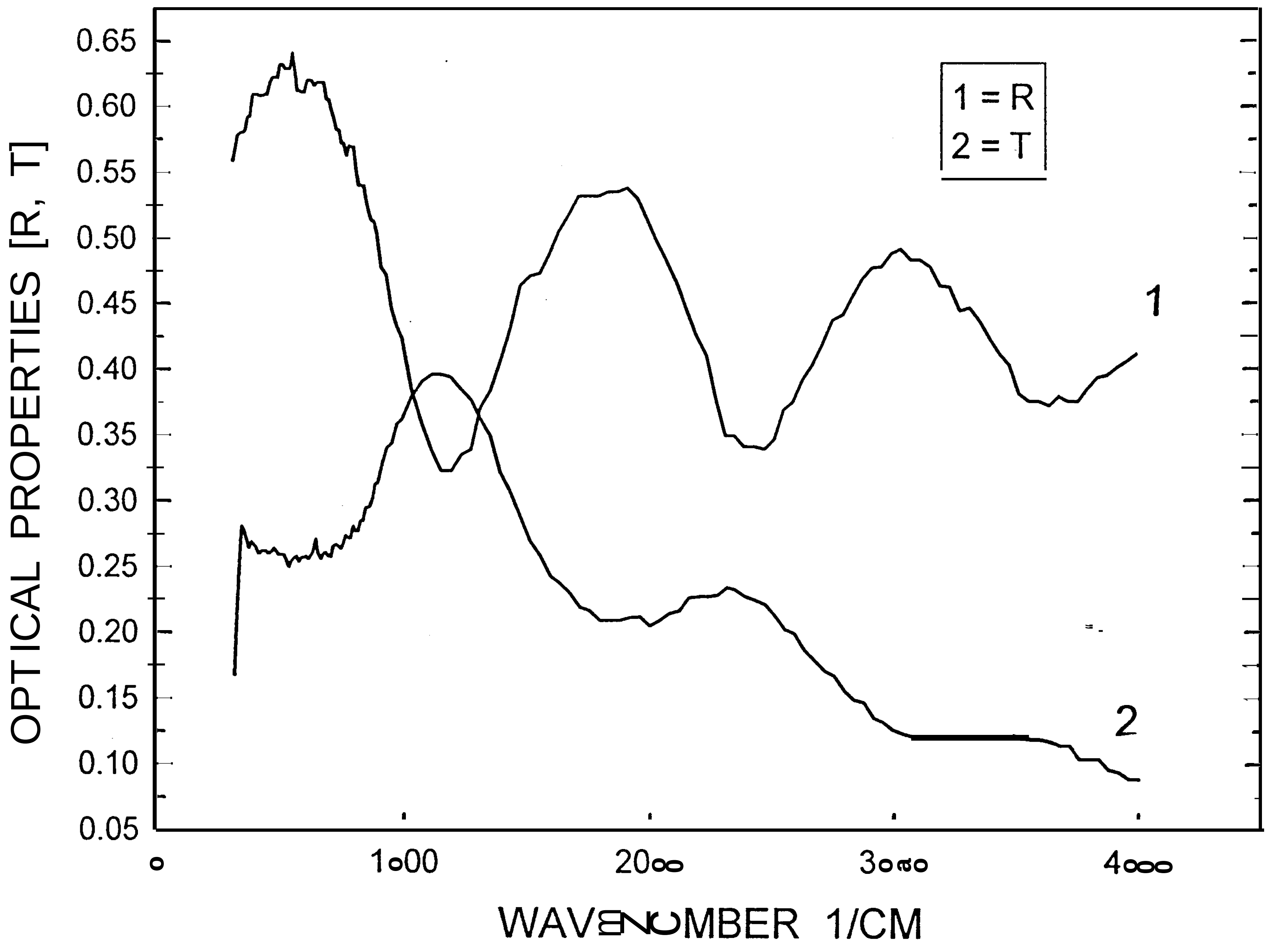


Fig 1b

Fig 1c

Fig. 2a



OPTICAL PROPERTIES [R, T]
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0
1000
2000
3000
4000
WAVENUMBER 1/CM
1 = R
2 = T
1
2

Fig. 2b

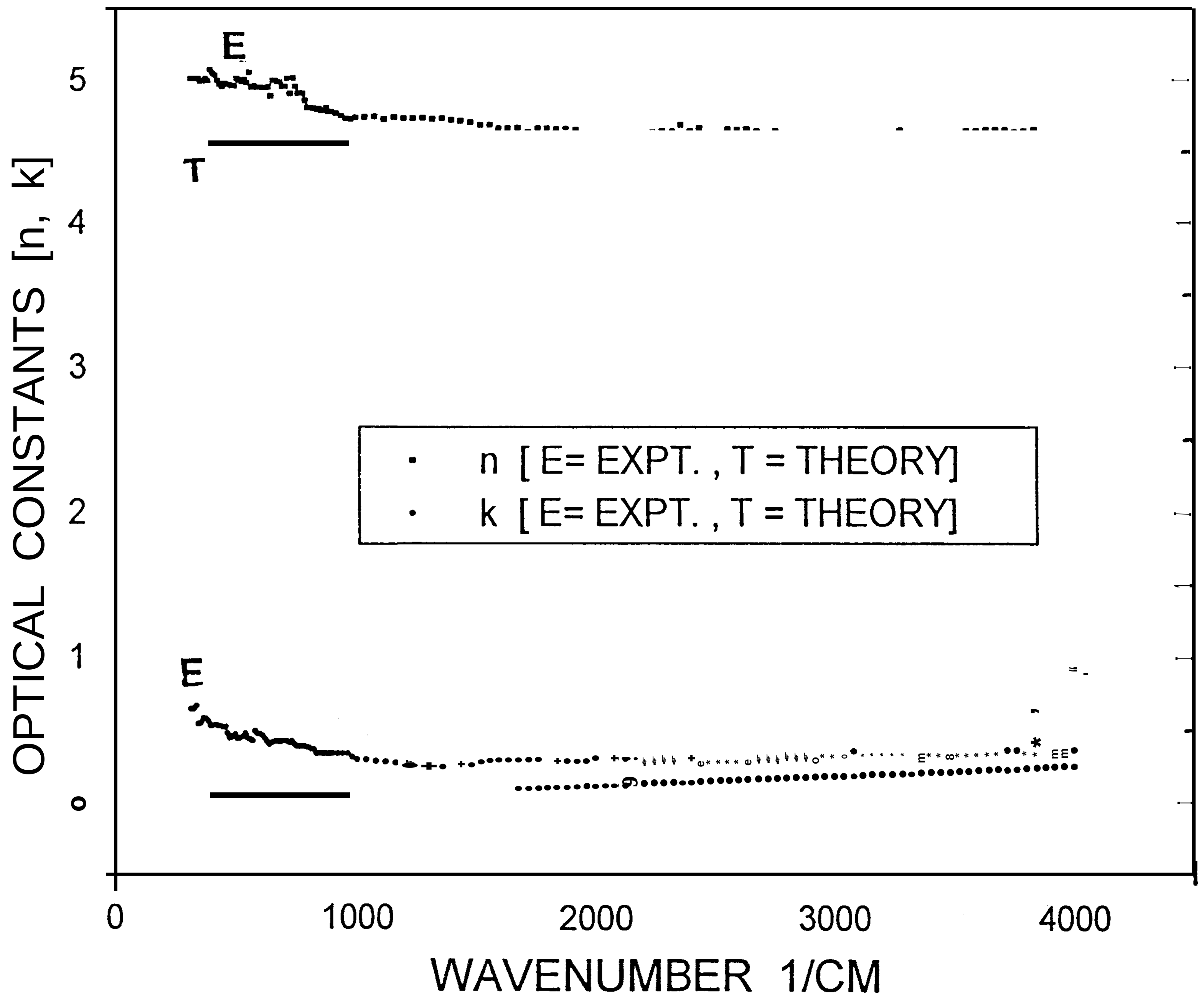
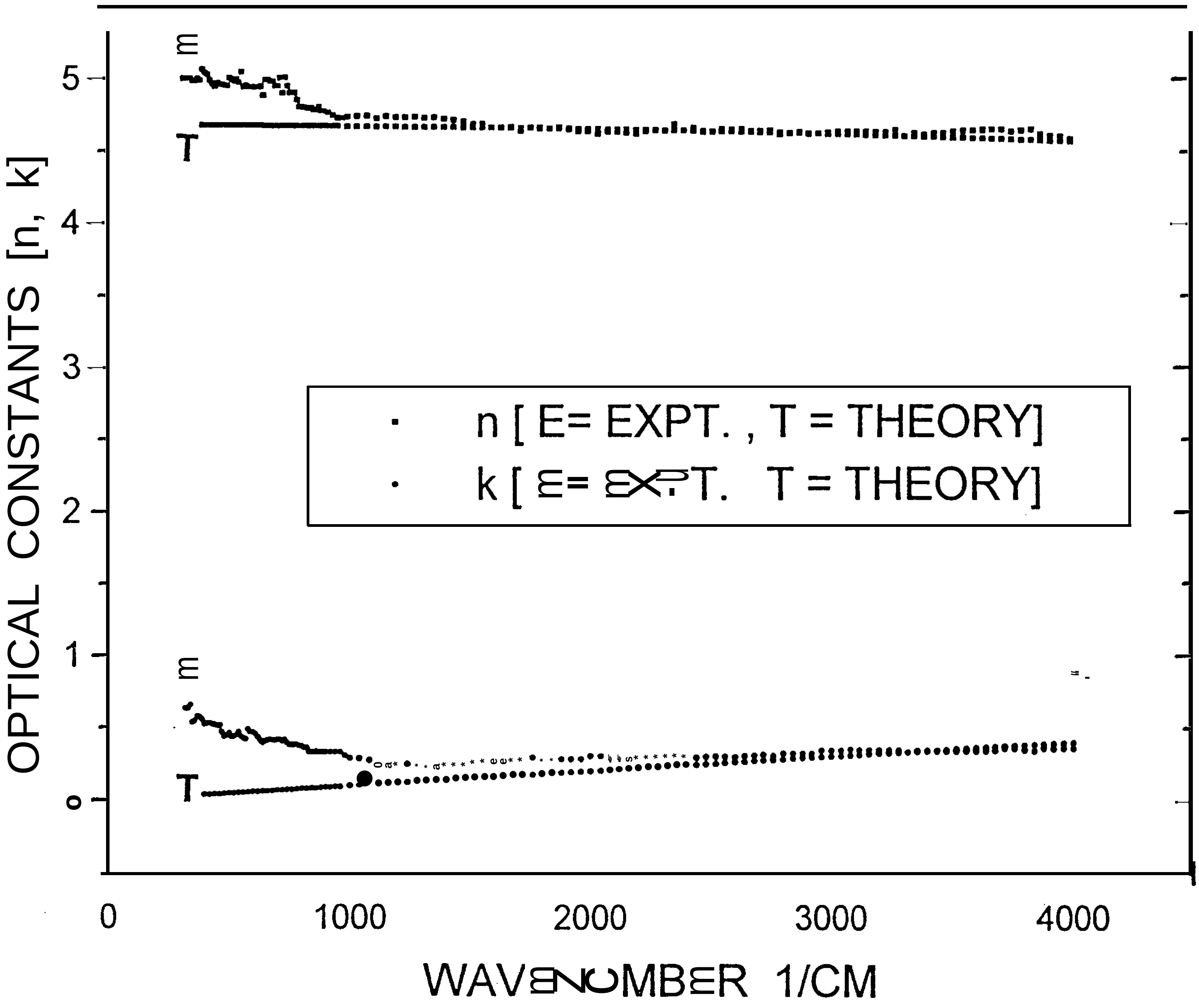
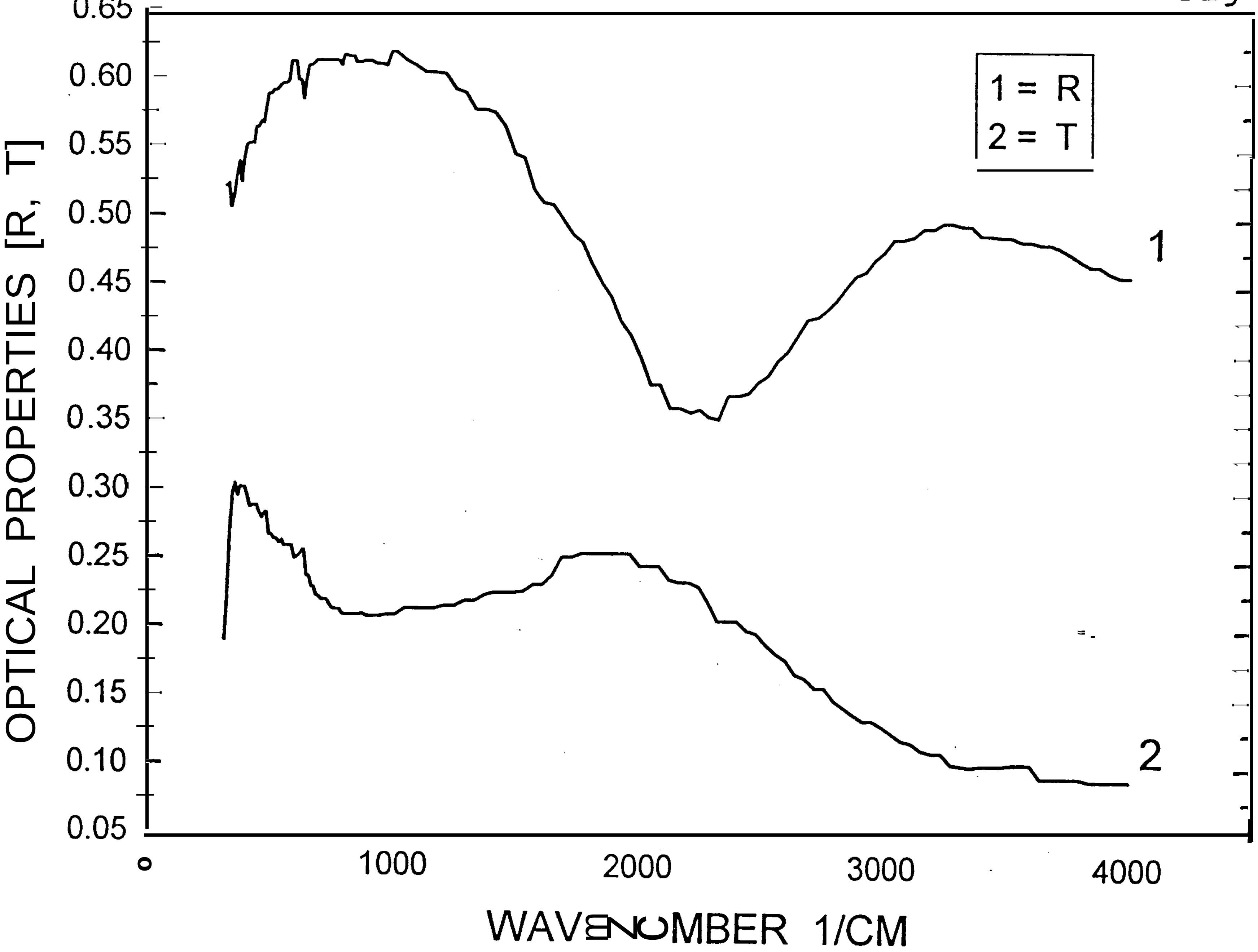

OPTICAL CONSTANTS [n, k]
5
4
3
2
1
o
E
T
E
n [E= EXPT. , T = THEORY]
k [E= EXPT. , T = THEORY]
0
1000
2000
3000
4000
WAVENUMBER 1/CM

Fig. 2c



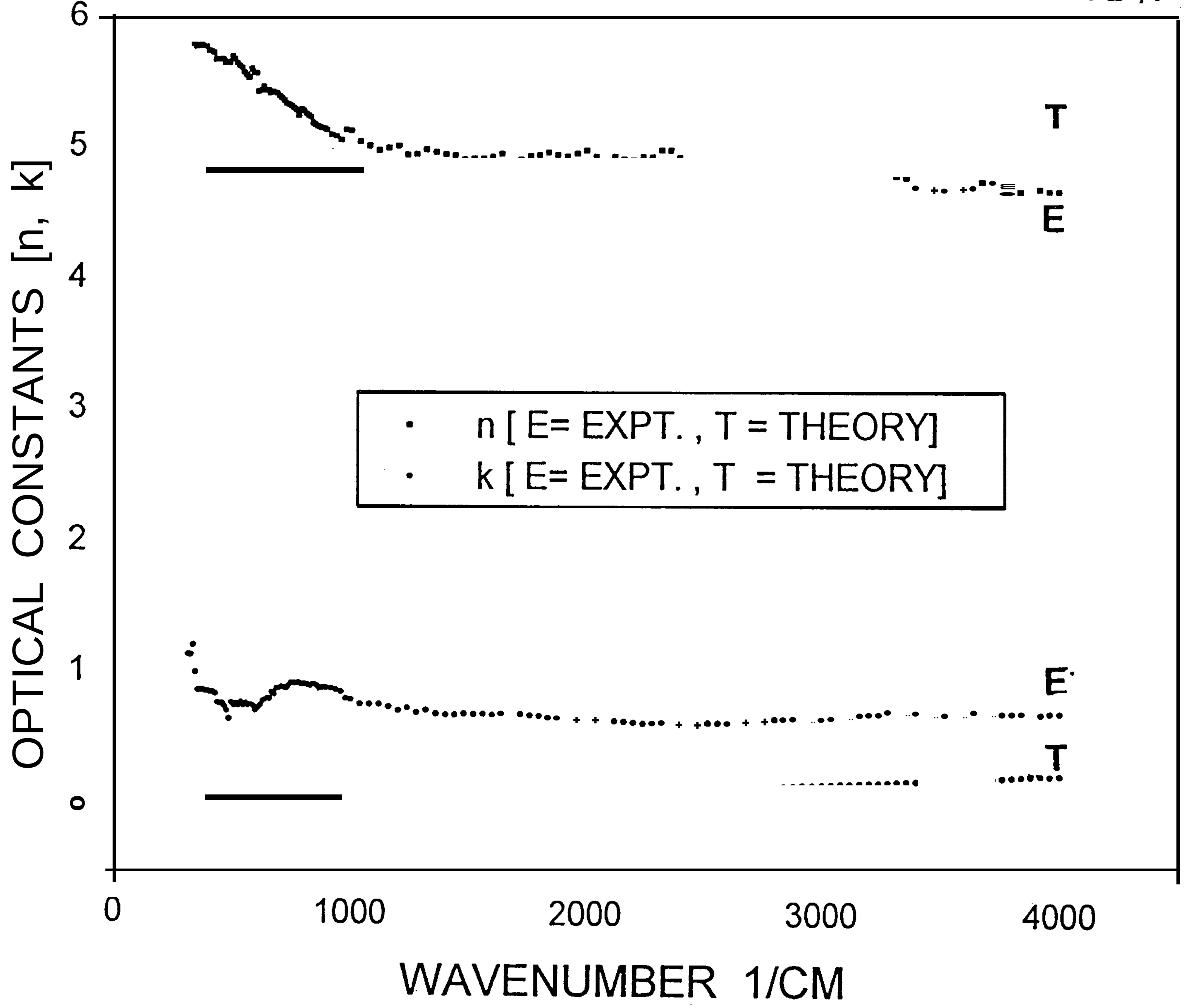
OPTICAL CONSTANTS [n, k]
5
4
3
2
1
0
E
T
n [E= EXPT. , T = THEORY]
k [E= EXPT. T = THEORY]
0
1000
2000
3000
4000
WAVENUMBER 1/CM

Fig. 3a



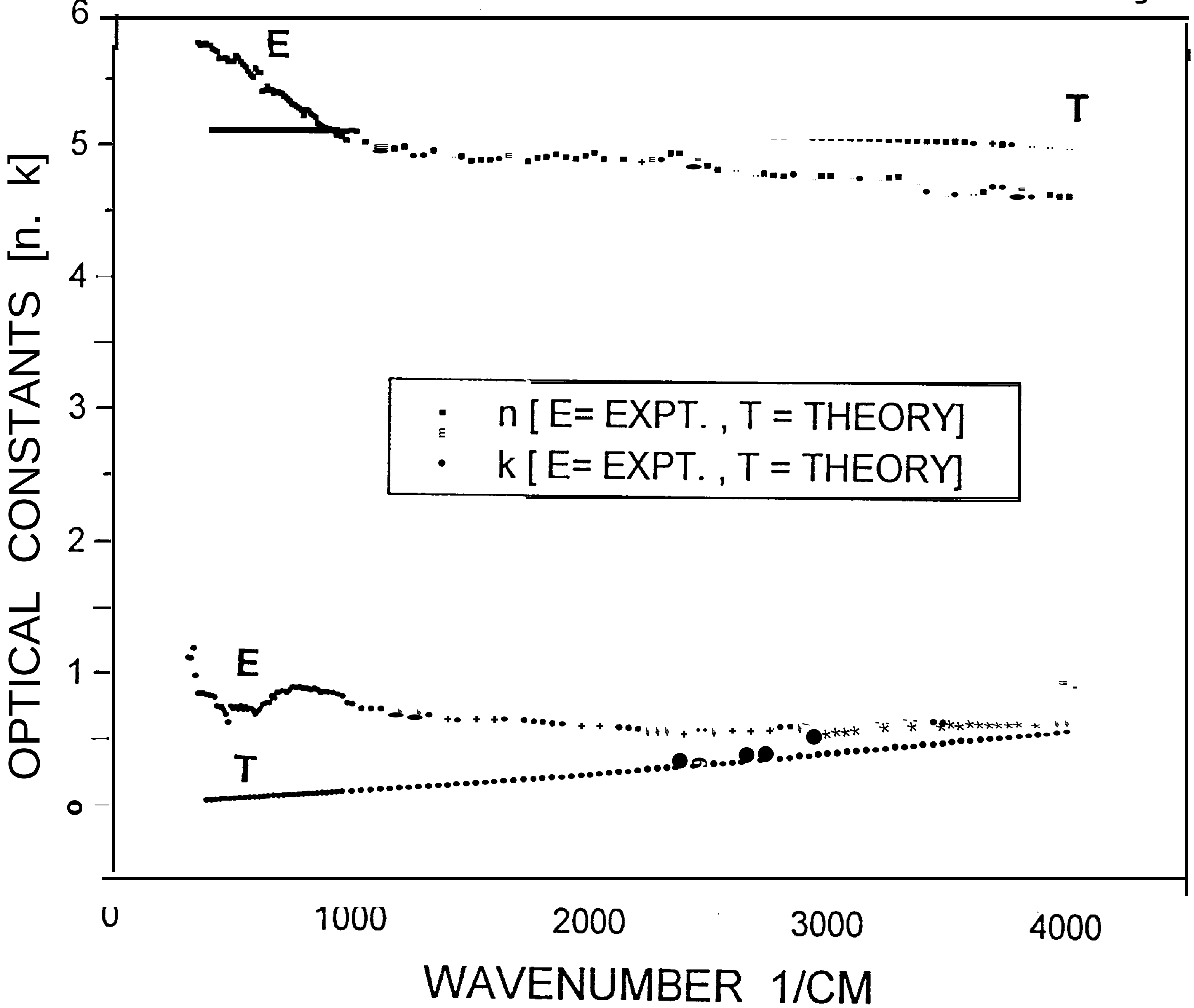
OPTICAL PROPERTIES [R, T]
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0
1000
2000
3000
4000
WAVENUMBER 1/CM
1 = R
2 = T
1
2

Fig. 3b



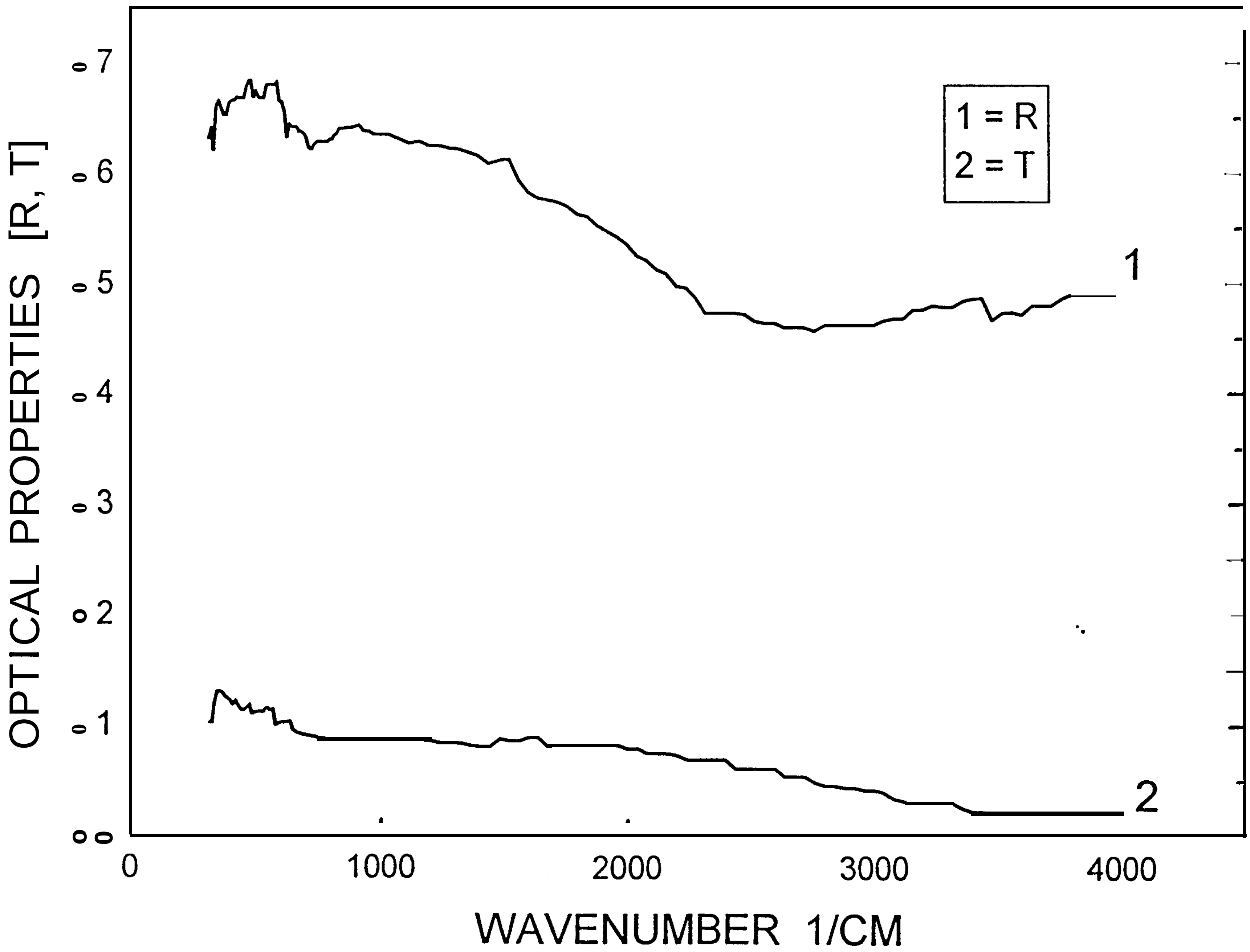
OPTICAL CONSTANTS [n, k]
6
5
4
3
2
1
o
0
1000
2000
3000
4000
WAVENUMBER 1/CM
n [E= EXPT. , T = THEORY]
k [E= EXPT. , T = THEORY]
T
E
E
T

Fig. 3c



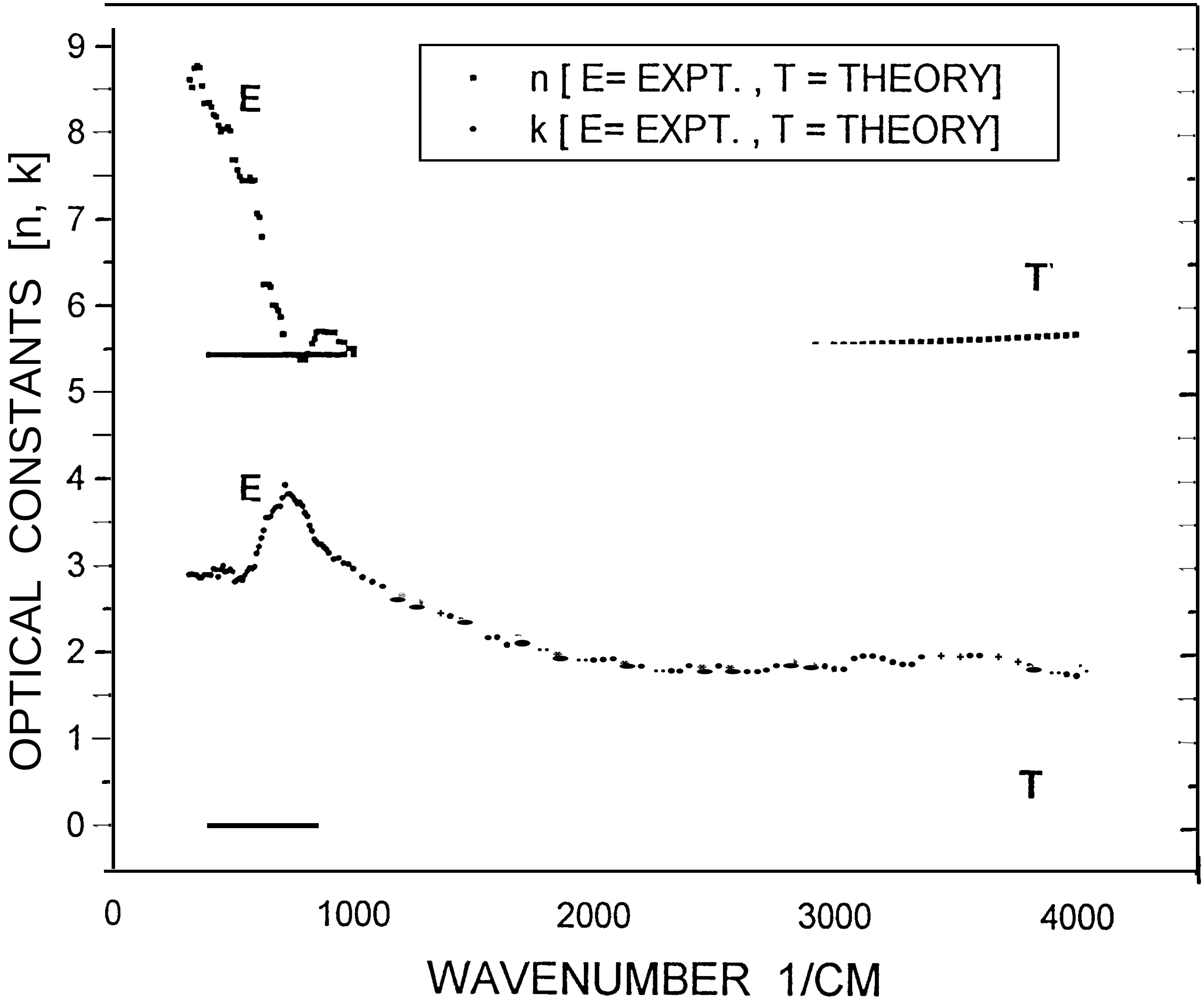
OPTICAL CONSTANTS [n. k]
WAVENUMBER 1/CM
0
1
2
3
4
5
6
0
1000
2000
3000
4000
E
T
E
T
n [E= EXPT. , T = THEORY]
k [E= EXPT. , T = THEORY]

Fig. 4a



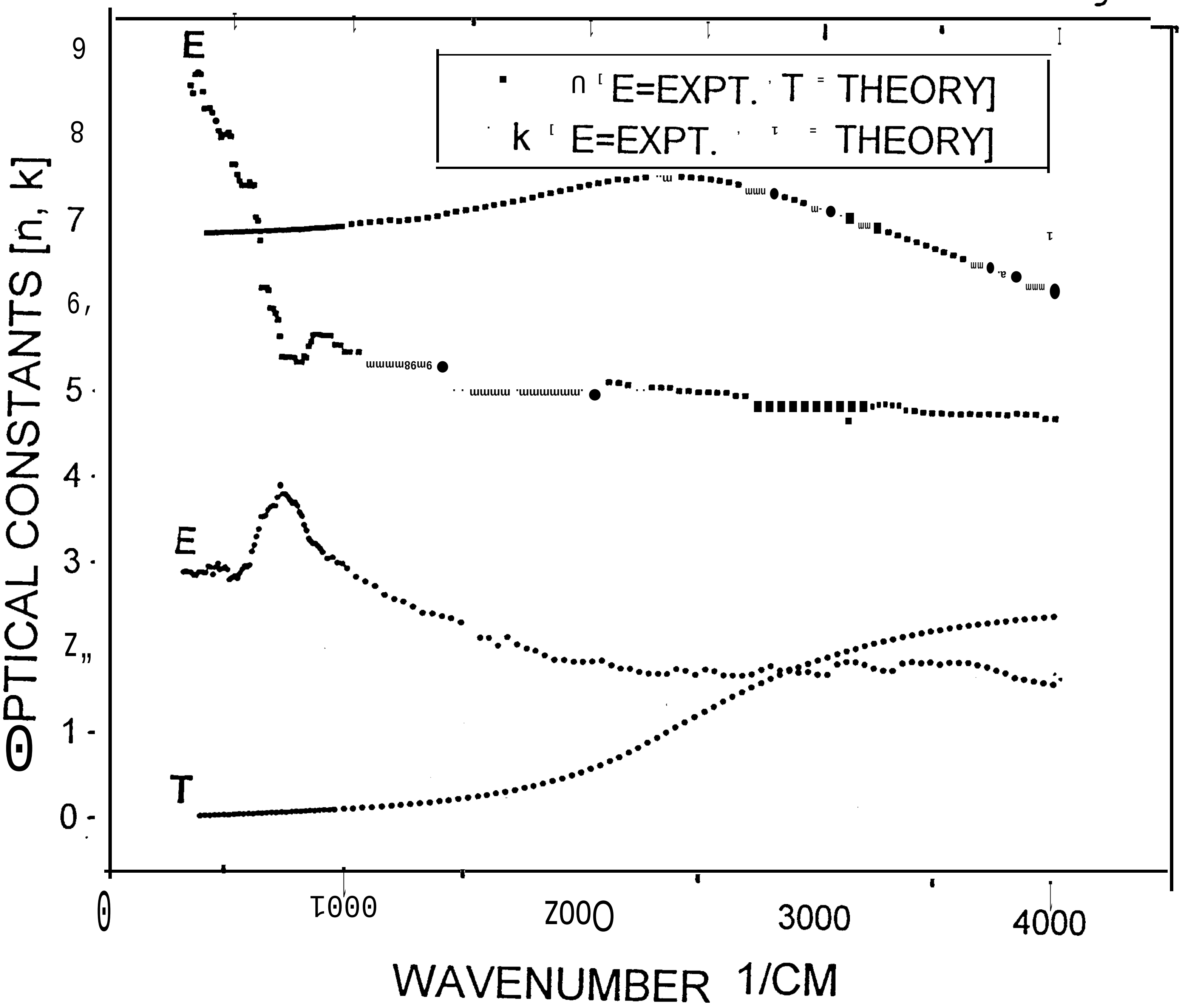
OPTICAL PROPERTIES [R, T]
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
0
1000
2000
3000
4000
WAVENUMBER 1/CM
1 = R
2 = T
1
2

Fig. 4b

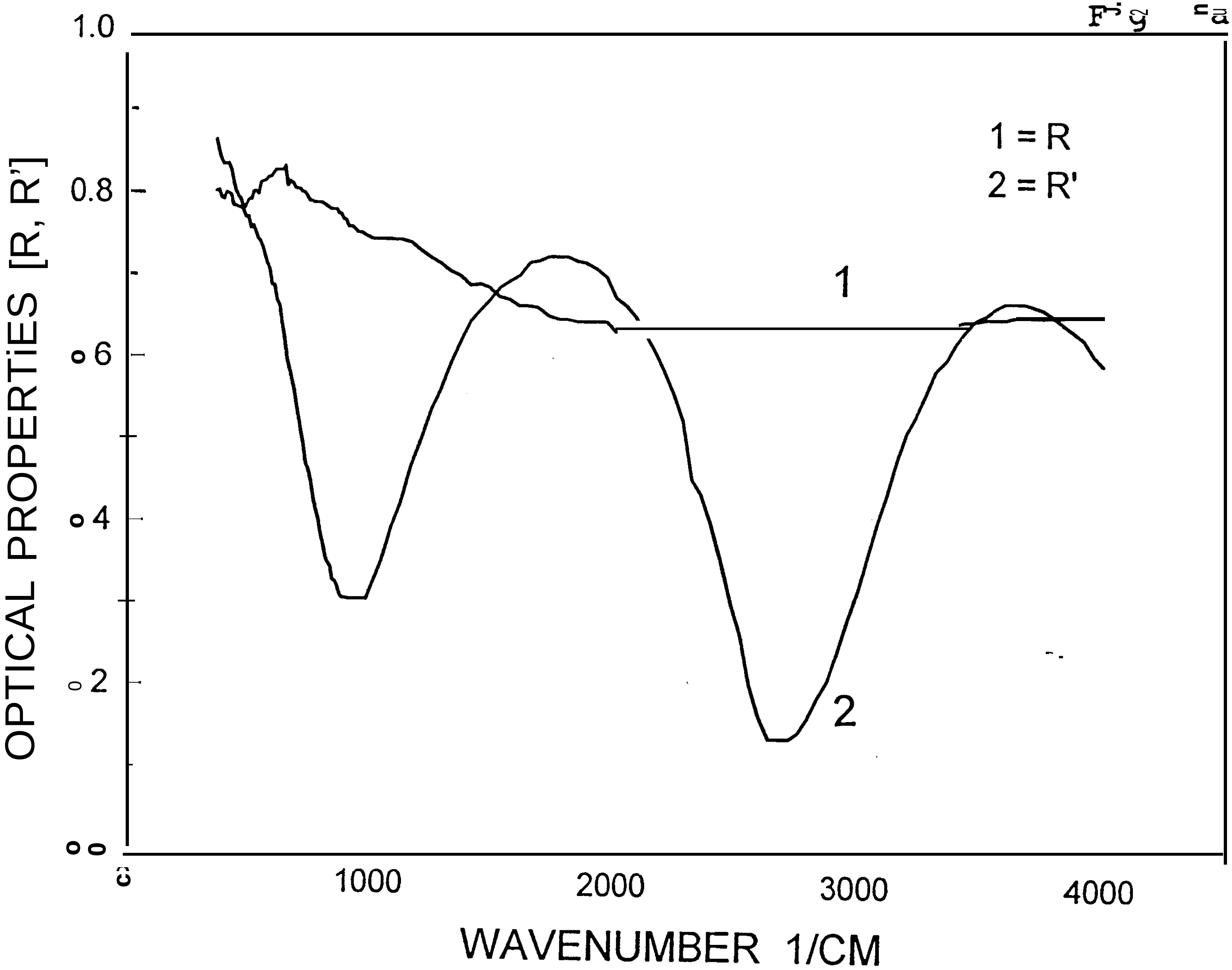


n [E= EXPT. , T = THEORY]
k [E= EXPT. , T = THEORY]
E
E
T
T
OPTICAL CONSTANTS [n, k]
0
1
2
3
4
5
6
7
8
9
0
1000
2000
3000
4000
WAVENUMBER 1/CM

Fig. 4c

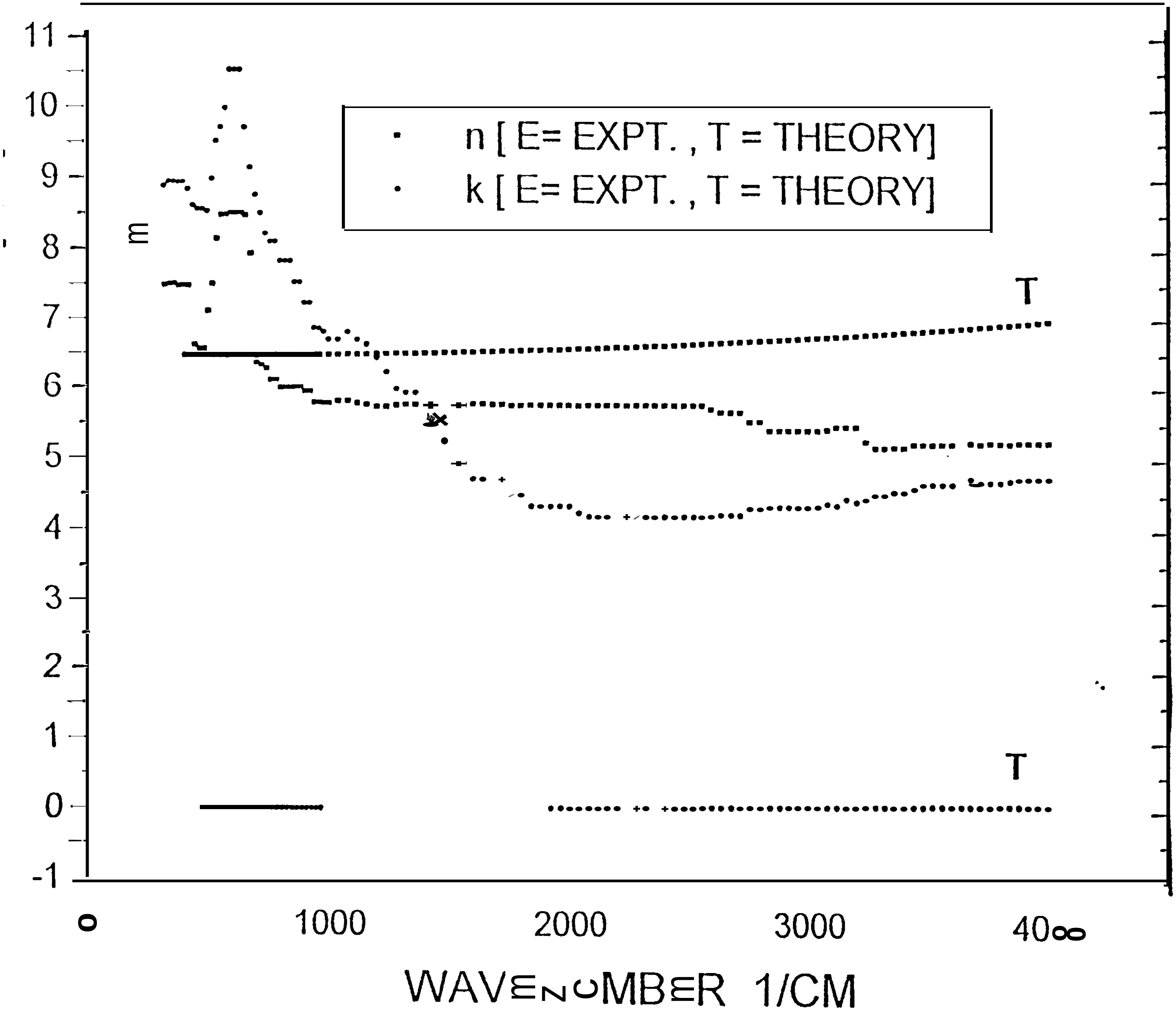


n [E=EXPT. T = THEORY]
k [E=EXPT. = THEORY]
E
E
T
OPTICAL CONSTANTS [n, k]
WAVENUMBER 1/CM
0
1000
2000
3000
4000
0
1
2
3
4
5
6
7
8
9



1.0
0.8
0.6
0.4
0.2
0.0
1 = R
2 = R'
1
2
1000
2000
3000
4000
OPTICAL PROPERTIES [R, R']
WAVENUMBER 1/CM

Fig. 5b



n [E= EXPT. , T = THEORY]
k [E= EXPT. , T = THEORY]
E
T
T
11
10
9
8
7
6
5
4
3
2
1
0
-1
0
1000
2000
3000
4000
WAVENUMBER 1/CM

Fig. 5c

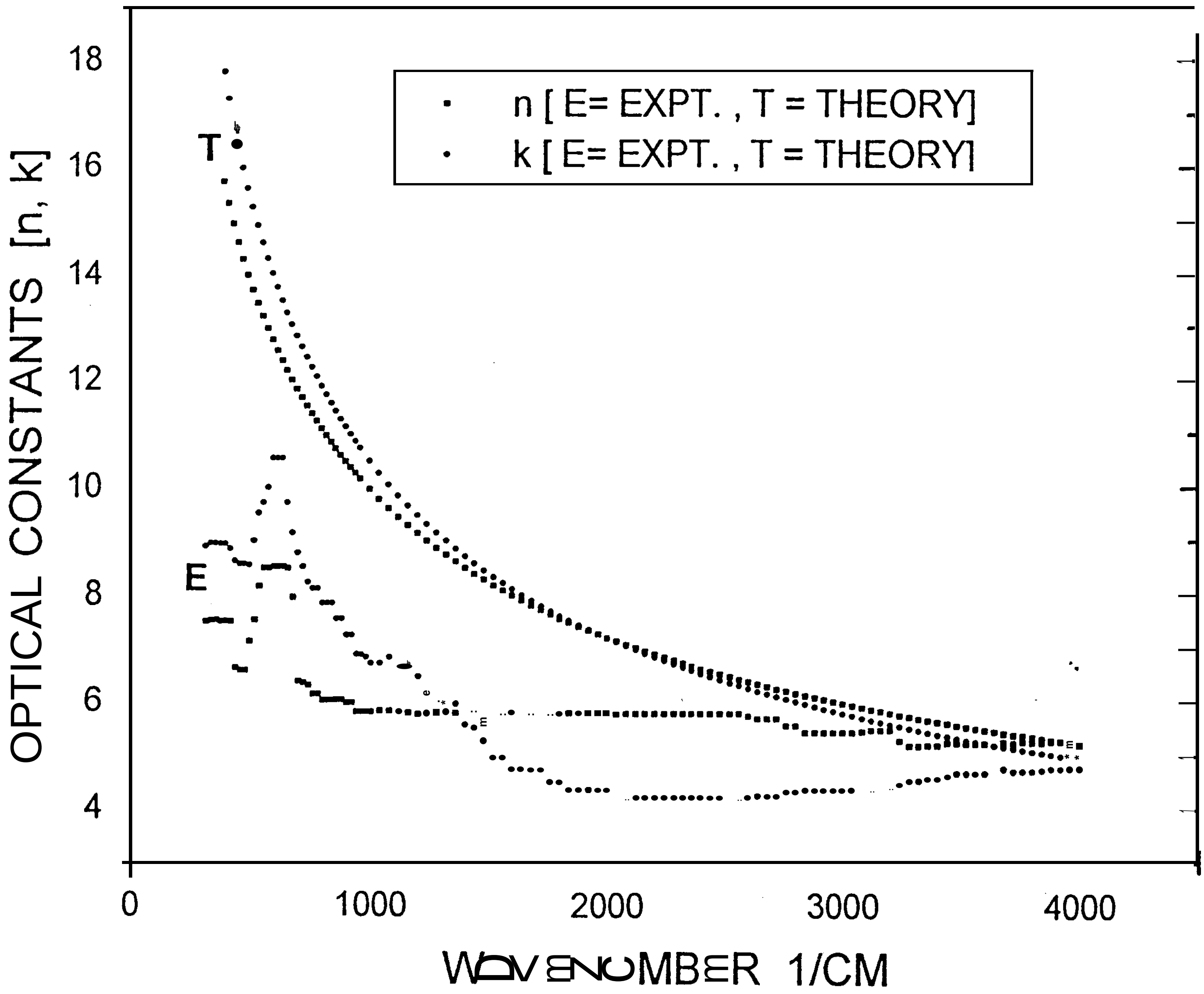
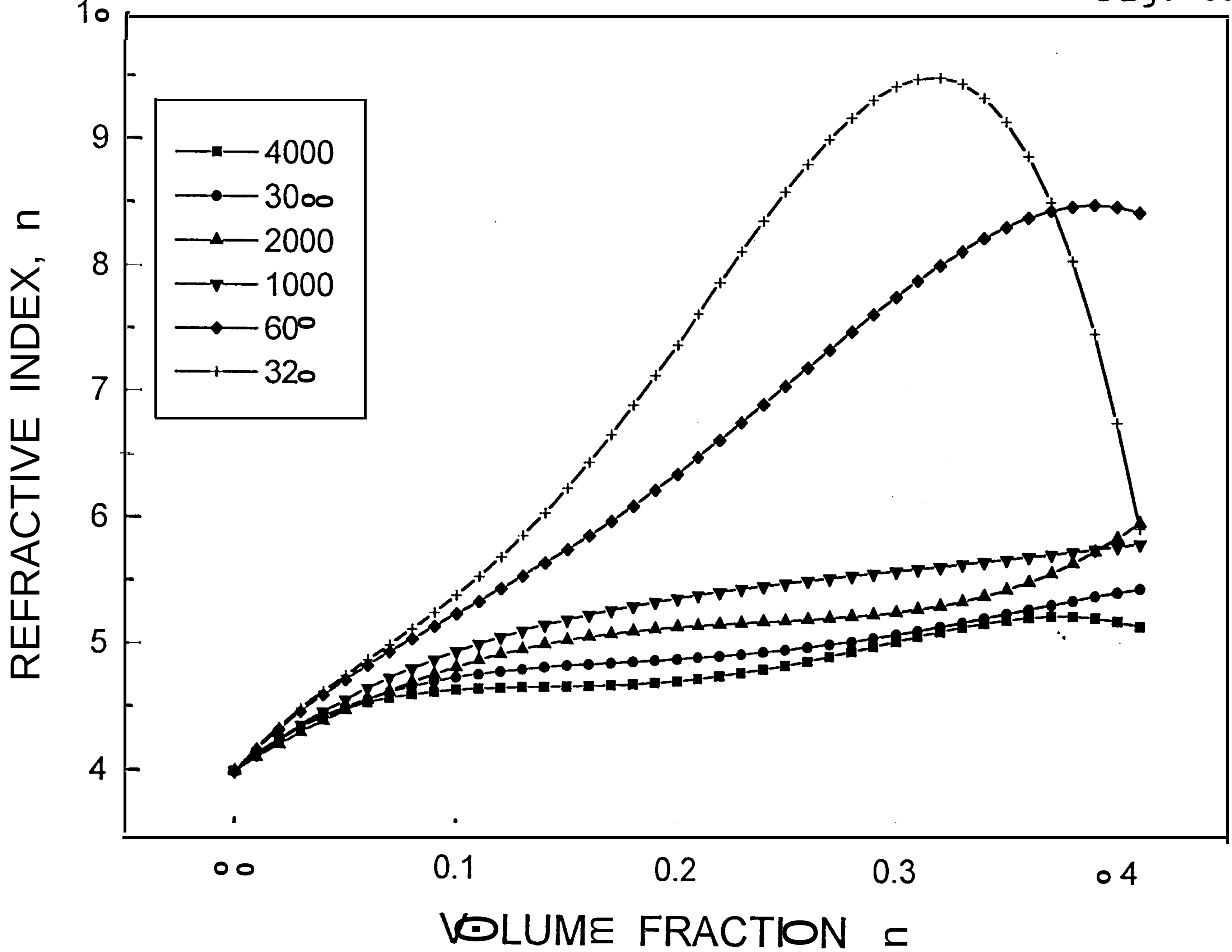
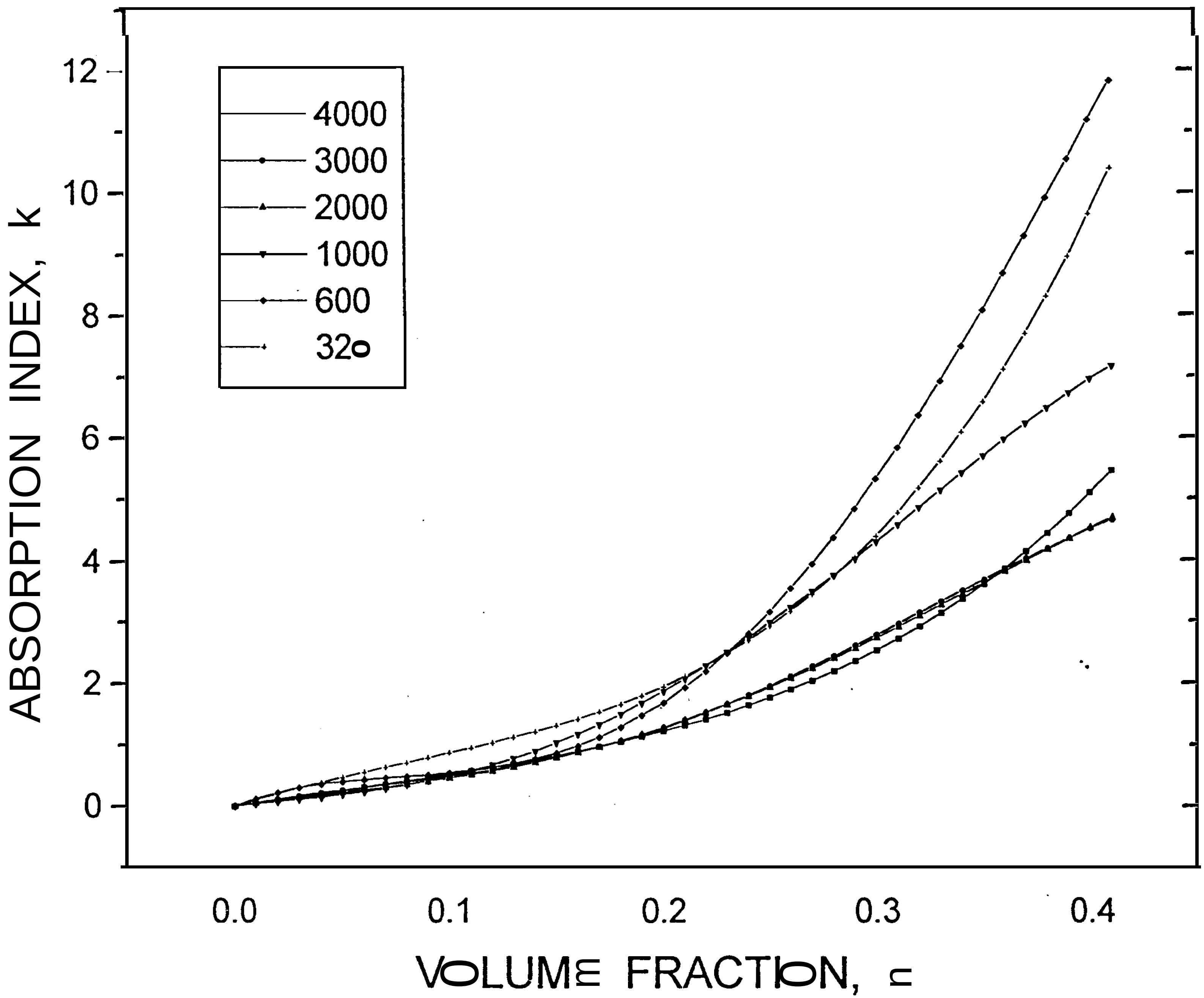

n [E= EXPT. , T = THEORY]
k [E= EXPT. , T = THEORY]
T
E
OPTICAL CONSTANTS [n, k]
18
16
14
12
10
8
6
4
0
1000
2000
3000
4000
WAVENUMBER 1/CM

Fig. 6a



REFRACTIVE INDEX, n
10
9
8
7
6
5
4
4000
3000
2000
1000
600
320
0.0
0.1
0.2
0.3
0.4
VOLUME FRACTION n

Fig. 6b


ABSORPTION INDEX, k
12
10
8
6
4
2
0
4000
3000
2000
1000
600
320
0.0
0.1
0.2
0.3
0.4
VOLUME FRACTION, n